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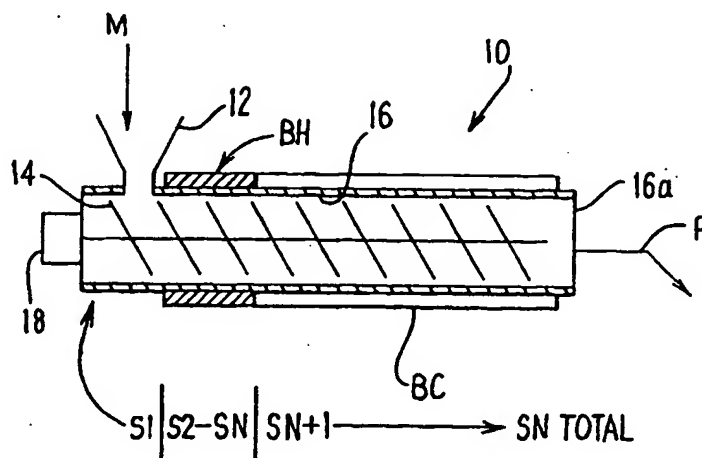
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(54) Title: **RECONSTITUTED POLYMERIC MATERIALS**



(57) Abstract

A method of making polymeric particulates (P) wherein polymeric scrap material, virgin polymeric material and mixtures thereof (M) are supplied to intermeshing extruder screws (14) which are rotated to transport the polymeric material along their length (SN Total) and subject the polymeric material to solid state shear pulverization and in-situ polymer compatibilization, if two or more incompatible polymers are present. Uniform pulverized particulates are produced without addition of a compatibilizing agent. The pulverized particulates are directly melt processable (as powder feedstock) and surprisingly yield a substantially homogenous light color product.

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## RECONSTITUTED POLYMERIC MATERIALS

## CONTRACTUAL ORIGIN OF THE INVENTION

This invention was made with Government support under Grant No. DE-FG 51-92 R 020215 awarded by the U.S. Department of Energy, Innovative Concepts Program, 392240.

## FIELD OF THE INVENTION

10 The present invention relates to solid state shear pulverization of polymeric material, which may include thermodynamically incompatible polymers, to form without compatibilizing agents pulverized particulates that are directly melt processable as powder feedstock to shaped articles of manufacture by conventional blow molding, rotational molding, extrusion, and spray coating techniques without color streaking in the resulting articles of manufacture.

## BACKGROUND OF THE INVENTION

20 Decreasing landfill space and rapidly rising disposal costs have forced many municipalities to begin curbside recycling of post-consumer plastic (polymeric) waste. In general, plastic materials comprise approximately 20% by volume of the municipal waste stream. For example, Chem Systems, 1992, reports that municipal solid waste comprises, by weight, 48% polyethylene (PE) (27% being low density PE and 21% being high density PE), 16% polypropylene (PP), 16% polystyrene (PS), 6.5% polyvinyl chloride (PVC), 5%

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polyethylene terephthalate (PET), 5% polyurethane, and 3.5% other plastics.

Post-consumer polymeric waste, as opposed to industrial plastic waste, typically includes substantial quantities of plastic bottles, containers and packaging materials. Plastic bottles are molded of different polymeric materials depending upon the product they are to contain. For example, plastic bottles for water, milk, and household chemicals typically are made of high density polyethylene (HDPE), while soft drink bottles are typically made of polyethylene terephthalate (PET) with or without base caps made from high density polyethylene (HDPE). Generally, HDPE bottles account for approximately 50-60% and PET bottles account for approximately 20-30% of the bottles used by consumers. The balance of bottles, bottle caps and other containers used by consumers comprises other polymeric materials, such as low density polyethylene (LDPE), polypropylene (PP), polystyrene (PS), polyvinyl chloride (PVC), and other resins and multi-layered materials.

Plastic packaging materials also are made of a wide variety of polymers. For example, according to Plastics Compounding, Nov/Dec, 1992, the following polymers were used in packaging material in the %'s set forth: 27% LDPE, 21% HDPE, 16% PS, 16% PP, and 5% PET.

Post-industrial plastic waste can comprise polyolefins, PS, PET and other polymeric materials used for plastic packaging.

Currently, collection of plastic waste material exceeds the market demand for recycled plastic products as a result of the dearth of viable recycling technologies that are low cost and produce high quality recycled plastic products. One recycling approach has involved the high energy consuming batch grinding of commingled, unsorted mixed color plastic waste to form flake scrap material, melt processing and pelletizing

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the melt processed material to pellets, and extruding the pelletized plastic waste to form recycled plastic products. However, recycled plastic products made in this manner suffer from severe deficiencies that render the products unsatisfactory for many purposes and are of inferior, low value compared to products made of virgin polymeric materials. For example, these recycled plastic products exhibit inferior mechanical properties (e.g. tensile, flexural and impact strength) and inferior appearance in terms of color (dark brown or gray color) with streaking of colors within the molded product as a result of the chemical incompatibility of the different polymers present in the initial plastic waste stream and variations in the plastic waste stream composition over time.

A typical example of a low value, recycled plastic product is recycled plastic lumber having a dark brown or gray color with noticeable color streaking and inferior mechanical properties compared to components molded of virgin materials. As a result of the less than pleasing appearance, recycled plastic lumber is oftentimes painted to improve its appeal to the customer, or expensive pigments and other additives are added to the feedstock during the manufacturing process to this end. However, the cost of the recycled product is increased thereby.

Furthermore, certain melt processing techniques, such as blow molding, rotational molding, extrusion (e.g. extruded PVC pipe and profiles), and spray coating, require a plastic powder feedstock. That is, the flake scrap material is not directly melt processable to articles of manufacture by such powder feedstock-requiring melt processing techniques. To be useful as feedstock in such melt processing techniques, sorted or unsorted flake scrap material produced by batch grinding must be pelletized and then ground to powder form.

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The need to pelletize and grind sorted or unsorted flake scrap polymeric material prior to such melt processing adds considerably to the cost and complexity of recycling scrap plastics as well as the capital equipment expenditures required.

10       Currently used injection molding techniques require plastic pellets for high speed production of molded parts. Although unsorted, commingled flake scrap materials could be pelletized to provide feedstock for injection molding, the resultant molded products would suffer from the types of deficiencies discussed above attributable to polymer incompatibility.

20       So-called compatibilizing agents and/or reinforcing agents can be added to flake plastic scrap material comprising chemically incompatible polymers in attempts to produce a recycled plastic product exhibiting more desirable characteristics. However, addition of these agents to the plastic scrap material makes recycling more difficult and adds considerably to its cost. The Mavel et al. U.S. Patent 4 250 222 relates to this type of recycling approach and is representative of the disadvantages associated with such an approach to plastic recycling.

30       Attempts have been made to sort commingled, post-consumer plastic scrap to overcome the polymer incompatibility problems associated with the recycling of commingled plastic scrap. To-date, HDPE and PET are recovered from plastic waste streams by recycling technologies requiring sorting of the commingled plastic materials. Sorting can require use of costly techniques, such as video cameras, electronic devices, infrared detectors, and organic "markers", to provide effective segregation of like plastics. However, even sorted plastic waste can present problems in processing as a result of density and chemical differences among

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polymers falling in the same general class and made by different plastics manufacturers.

Further, sorted plastic scrap must be subjected to batch grinding to produce flake scrap material that then must be pelletized and ground again to provide powder feedstock for blow molding, rotational molding, some extruding, spray coating and other melt processing techniques that require powder feedstock.

10 The high cost of sorting has greatly limited widespread use of recycling approaches that require a sorting step. In particular, collected and sorted post-consumer plastic materials are usually more expensive than the corresponding virgin plastic materials. Thus, users of plastic materials are discouraged from using sorted, recycled plastic materials.

20 It is an object of the invention to provide a method of processing one or more polymeric materials, such as sorted or unsorted, commingled scrap polymeric material, by solid state pulverization to produce pulverized particulates (e.g. powder) that can be directly formed to shape by powder feedstock-using melt processing techniques.

30 It is another object of the invention to provide a method of processing polymeric materials, such as sorted or unsorted, commingled scrap polymeric materials, having mixed colors by solid state pulverization to produce pulverized particulates that are melt processable to a substantially homogeneous light color without color streaking or marbleizing despite being produced from the mixed color scrap materials.

It is a further object of the invention to provide a method of processing polymeric materials in a manner to achieve in-situ compatibilization of different polymers present.

It is a further object of the present invention to provide a method of recycling commingled scrap polymeric

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materials without sortation and in a manner to achieve in-situ compatibilization of different polymers present and produce recycled polymeric particulates without the need for a compatibilizing agent.

It is still another object of the present invention to provide a method of recycling commingled, mixed-color scrap polymeric materials without sortation and in a manner to produce recycled polymeric particulates that are melt processable to homogeneous light color without color streaking or marbleizing.

It is still another object of the present invention to provide solid state pulverized polymeric particulates that are suitable as powder feedstock for melt processing by blow molding, rotational molding, some extruding, spray coating and other powder feedstock-using melt processing techniques.

It is still a further object of the present invention to provide solid state pulverized polymeric particulates that are melt processable to a homogenous light color, despite being produced from mixed-color polymers.

It is still a further object of the invention to produce articles of manufacture, including molded parts and coatings, made from the aforementioned solid state pulverized polymeric particulates.

#### SUMMARY OF THE INVENTION

The present invention provides in one aspect a method of making polymeric particulates (e.g. powder) wherein sorted or unsorted, commingled polymeric scrap material, virgin polymeric material and mixtures thereof are supplied to extruder screw means rotated to transport the material along the length thereof and in the solid state convert the material to pulverized particulates (e.g. powder) that are melt processable directly by conventional blow molding, rotational molding, extrusion, spray coating and other melt



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processing techniques requiring a powder feedstock. This avoids the need for and costs associated with flake pelletizing and pellet grinding operations heretofore required.

The solid state pulverized particulates also are melt processable by conventional molding, extruding, spray coating and the like to form articles of manufacture having a substantially homogenous color appearance without color streaking or marbling. This color homogeneity is achievable regardless of whether the particulates include mixed color polymeric material of the same or different composition. This avoids the need for the addition of pigments and/or compatibilizing agents to the feedstock and the need to paint the molded or extruded product to hide unpleasing colors and color streaking.

The present invention provides in another aspect a method of making polymeric particulates wherein polymeric material, such as unsorted polymeric scrap material, comprising two or more thermodynamically incompatible polymers is supplied to extruder screw means rotated to transport the material along the length thereof and subject the material to solid state pulverization and in-situ polymer compatibilization. In-situ polymer compatibilization is evidenced, in one instance, by the resulting pulverized polymeric particulates exhibiting a thermogram different from that of the precursor unpulverized material. For example, the pulverized particulates of the invention exhibit a melting peak and/or crystallization peak quite different from that (those) of the unpulverized material. Moreover, molded articles produced from the pulverized particulates of the invention exhibit increased tensile strengths and lack of delamination upon breaking in mechanical testing, this being a further indication of in-situ polymer compatibilization.

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In practicing the present invention, the polymeric scrap material and/or virgin material can include thermoplastics, polymer blends, polymer alloys, thermosets, elastomers and other polymeric materials. Typically, the polymeric material is comminuted to flake form by grinding, chopping or shredding using conventional equipment prior to pulverization. The pulverization process uses as scrap feedstock a material that is in a physical form (e.g. comminuted flakes) commonly available from scrap collections and municipal recycling centers.

Also, in practicing the present invention, the polymeric material can be heated during the initial stage of the pulverization operation depending upon the make-up (composition) of the feedstock followed by cooling during subsequent stages of the pulverizing operation to maintain proper temperature control for solid state pulverization, in-situ polymer compatibilization and production of desired powder size. Preferably, however, the polymeric material is only subjected to frictional heating during the initial stage of the pulverization operation by engagement with the rotating screws. That is, solid state shear pulverization of the polymeric material preferably is conducted without heating of the material by any external extruder barrel heating device. Temperature control of the polymeric material during the pulverization operation is thereby facilitated to reduce degradation of the polymers and dye materials used with the feedstock polymers. Energy consumption during the pulverization operation also is reduced.

The present invention provides in still another aspect a method of making an article of manufacture having a substantially homogenous color from mixed-color polymeric material, such as sorted or unsorted, commingled polymeric scrap material. In this embodiment of the invention, mixed-color polymeric material of the

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same or different composition is supplied to extruder screw means rotated to transport the polymeric material along the length thereof to subject the material to solid state pulverization to form pulverized particulates. The pulverized particulates are molded, extruded or otherwise melt processed to form a substantially homogeneously colored shape characterized by the absence of color streaking and marbling, despite the particulates originating from mixed-color polymeric material. Typically, the pulverized powder is processable to a substantially homogenous pastel color tone corresponding to a dominant color of a particular scrap component in the feedstock.

The present invention also provides solid state pulverized particulates produced from scrap polymeric material and/or virgin polymeric material wherein the particulates are suitable as powder feedstock, without conventional melt pelletizing and pellet grinding, for direct melt processing to shape using blow molding, rotational molding, some extrusion, spray coating, and other powder feedstock-using techniques.

The present invention further provides solid state pulverized polymeric particulates comprising two or more otherwise thermodynamically incompatible polymers produced from commingled, unsorted polymeric scrap materials and/or virgin materials. The polymers are in-situ compatibilized by solid state shear pulverization as evidenced by one or more different thermogram characteristics between recycled particulates of the invention and unpulverized polymeric material. Typically, the solid state pulverized particulates exhibit enhanced reactivity as compared to the unpulverized polymeric material.

Moreover, the present invention provides solid state pulverized polymeric particulates that exhibit, pulverized and as-melt processed, a substantially

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homogenous color despite being pulverized from mixed-color scrap material.

Articles of manufacture and powder coatings produced from the solid state pulverized particulates of the present invention exhibit mechanical properties generally superior to those exhibited by like processed flake polymeric material of the same composition depending on the polymer components involved.

10                   Importantly, they also exhibit a substantially homogeneous color characterized by the absence of color streaking or marbleizing. Typically, the articles of manufacture exhibit a substantially homogeneous pastel color tone corresponding to a dominant color of a scrap component in the polymeric feedstock. Importantly, the recycled, pulverized particulates of the invention made from mixed-color polymeric feedstock can be used in molding a plurality of articles of manufacture that exhibit substantially the same homogeneous pastel color from one article to the next. In contrast, a mixture of  
20                   unpulverized flake polymeric material of like composition and mixed color produces molded articles exhibiting inconsistent colors from one molded article to the next.

30                   The present invention is advantageous in that the pulverized particulates are suitable for direct use as powder feedstock for powder feedstock-using melt processing techniques without the need for pelletizing and pellet grinding operations. Moreover, commingled scrap polymer materials, virgin polymeric materials and mixtures thereof can be processed in a manner to achieve in-situ compatibilization of different polymers in a once-through pulverization operation without the need for a compatibilizing agent and without sortation in the case of commingled scrap feedstock. The pulverized particulates may be mixed with fillers, reinforcing agents, flame retardants, antioxidants and other

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additives commonly used in the plastics industry if desired.

Moreover, the present invention is advantageous in that sorted or unsorted, commingled mixed-color polymeric materials and/or virgin polymeric materials can be pulverized as polymeric particulates that are melt processable to substantially homogeneous light color without the color streaking or marbling heretofore experienced using other recycling procedures.

10 The present invention can provide a high value, low cost recycled particulates product, as well as products molded or otherwise melt processed therefrom, thereby increasing utilization of available plastic scrap.

The aforementioned objects and advantages will become more readily apparent from the following detailed description and drawings.

#### BRIEF DESCRIPTION OF THE DRAWINGS

20 Figure 1 is a schematic sectional view of a twin-screw extruder for practicing an embodiment of the invention.

Figure 2 represents the morphological transformation of flake-shaped scrap feedstock to powder due to solid state shear pulverization in the extruder in accordance with the invention.

Figure 3 is an elevational view of a representative screw used in practicing the invention when the scrap material is heated by heater bands on the extruder barrel (partially shown).

30 Figure 4 is an elevational view of a representative screw used in practicing the invention when the scrap material is subjected only to frictional heat in the extruder barrel (partially shown).

Figures 5A, 6A, 7A and 8A are photographs of post-consumer, flake scrap feedstock and specimens injection molded therefrom and Figures 5B, 6B, 7B and 8B are

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photographs of pulverized powder of the invention and specimens injection molded therefrom.

Figures 9, 10, and 11 are electron spin resonance (ESR) spectra of various as-received post-consumer, flake scrap samples and pulverized powder samples of the invention of various compositions.

Figures 12A,B-18A,B are DSC (differential scanning calorimetry) thermograms of various as-received post-consumer, flake scrap samples and pulverized powder samples of the invention of various compositions.

#### DETAILED DESCRIPTION

In one embodiment, the present invention provides a method of making recycled polymeric particulates, such as powder, from post-consumer and/or post-industrial polymeric scrap material that may be sorted or unsorted, commingled so as to include two or more different scrap polymers.

Post-consumer polymeric waste typically includes substantial quantities of plastic bottles, containers and packaging materials made of different polymers. For example, plastic bottles for water, milk, and household chemicals typically are made of high density polyethylene (HDPE), while soft drink bottles are typically made of polyethylene terephthalate (PET) with or without base cups made of HDPE. Generally, HDPE bottles account for approximately 50-60% and PET bottles account for approximately 20-30% of the bottles used by consumers. The balance of bottles and other containers used by consumers comprise other polymeric materials, such as low density polyethylene (LDPE), polypropylene (PP), polystyrene (PS), polyvinyl chloride (PVC), and other resins and multi-layered materials.

Post-consumer polymeric waste also includes plastic packaging materials that are made of a wide variety of polymers including LDPE, HDPE, PS, PP, PET and others.

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Post-industrial plastic waste can comprise polyolefins, PS, PET, and other polymeric materials used for plastic packaging.

Sorted polymeric scrap material typically comprises a single polymer composition collected from one or more sources and sorted pursuant to standard polymer codes now used on plastic bottles, containers and the like. Sorted polymeric scrap material typically includes scrap of different colors since to-date color sorting is not yet common.

Unsorted, commingled scrap material can include myriad types of polymeric scrap materials including, but not limited to, HDPE, LDPE, PP, PS, PET, PVC, PC (polycarbonate), ABS/PC (acrylonitrile butadiene styrene/polycarbonate), PPO (polyphenylene oxide)/PS and others. In general, commingled, unsorted scrap can include thermoplastics, polymer blends, polymer alloys, thermosets, elastomers and other scrap polymeric materials.

Typically, unsorted, commingled scrap material will include mixtures of incompatible polymers such as mixtures of HDPE and PET, mixtures of HDPE and PS, mixtures of PP and PS, mixtures of HDPE, LDPE with PET and/or PS for purposes of illustration. By mutually thermodynamically incompatible is meant that two or more polymers have different glass transition temperatures,  $T_g$ , and when processed by melt mixing, separate into distinct microscopic phases which are reflected in mechanical property deficiencies.

The presence of these incompatible polymers has rendered previous attempts at reclamation of unsorted, commingled scrap highly costly due to the need for sortation and/or addition of one or more of compatibilizing agents, reinforcing agents, fillers, and pigments and also ineffective in yielding a high value recycled product having aesthetic characteristics and mechanical

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properties approaching those achievable with virgin plastic material.

The commingled, unsorted plastic scrap typically will include polymeric scrap materials having various colors as a result of the wide variety of colorants used in the plastic bottling, container and other plastic industries.

10 Thermodynamic polymer incompatibility readily manifests itself in resulting molded polymeric products that are characterized by one or more of delamination, brittleness, and inconsistent mechanical properties from one molded part to the next. Moreover, parts molded from mutually incompatible, mixed-color polymers are typically characterized by inconsistent color from one molded part to the next and also by color streaking or marbleizing through the same molded part. These characteristics are observed regardless of whether the thermodynamically incompatible polymers are virgin materials or scrap polymer materials.

20 In accordance with one particular illustrative embodiment of the present invention, sorted plastic scrap material comprising a single polymeric composition is solid state shear pulverized in a once-through operation to produce recycled, polymeric particulates (e.g. powder) that are formable to shape by powder feedstock-using melt processing techniques without the need for pelletizing. The sorted scrap material can include polymers of the same composition or type and yet the same or different colors. The pulverized part-  
30 iculates of the invention are melt processable to substantially homogeneous light color without color streaking or marbleizing experienced heretofore with other recycled techniques.

In accordance with another particular illustrative embodiment of the present invention, unsorted, commingled plastic scrap materials are solid state shear



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pulverized to produce recycled, polymeric particulates (e.g. powder) without the need for costly sortation, without the need for pelletization prior to use as feedstock in powder feedstock-using melt processing techniques, and in a manner to achieve in-situ compatibilization of thermodynamically incompatible polymers in a once-through pulverization operation that avoids the need to add a compatibilizing agent.

Moreover, commingled, unsorted plastic scrap containing mixed-color scrap polymeric materials can be recycled without sortation to produce recycled, polymeric particulates (e.g., powder) melt processable to substantially homogeneous light color articles of manufacture without the color streaking or marbleizing heretofore experienced with other recycling procedures.

As used herein, the term color is intended to have a broad meaning to include usual color hues and white as well as transparent and translucent appearance.

As will become apparent herebelow, the recycled, polymeric pulverized particulates of the invention produced from sorted or unsorted, commingled scrap materials underwent through chemical changes as characterized by DSC (differential scanning calorimetry) and ESR (electron spin resonance spectroscopy) which features are dramatically different from the those exhibited by unpulverized flake scrap material of the same composition. Moreover, molded components produced from the pulverized particulates of the invention generally exhibit increased tensile strengths and lack of delamination upon breaking in mechanical testing depending upon the polymer components involved, these characteristics being indicative of in-situ polymer compatibilization.

In practicing the aforementioned illustrative embodiments of the present invention, polymeric scrap material is collected from several recycling centers

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(e.g. municipal recycling facilities commonly known as MRF's and/or industrial recycling centers). The collected scrap material may be already sorted by polymer type. In this event, each polymer type can be individually pulverized in accordance with the invention.

10 On the other hand, the collected scrap material may be unsorted and as a result include two or more different polymers which may be thermodynamically incompatible. Moreover, scrap material collected from different centers typically will be at least partially intermixed with scrap material collected from other centers as a result of the usual collection, transportation, storage, and handling procedures for the scrap material prior to recycling. Unsorted, commingled scrap material can result from this situation.

20 The as-collected scrap material, whether of the sorted or unsorted, commingled type, typically is initially cleaned to remove unwanted contamination. Cleaning of the as-collected scrap material can be effected by water rinsing and/or cleaning solutions to remove contaminants, such as food residue, detergents, oil, and other contaminants. However, the need for and type of cleaning procedure used for initial cleaning of the as-collected scrap material will depend upon the degree and the type of contamination present on the scrap material. Relatively clean as-collected scrap material may not require any cleaning prior to pulverization.

30 Before or after cleaning, the as-collected, scrap material, whether of the sorted or unsorted, commingled type, initially is comminuted by grinding, chopping or shredding prior to pulverization to provide a polymeric scrap feedstock comprising flakes F. The flakes F typically have sharp, angular surfaces resulting from the comminution operation and usually have different

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colors with the number of colors present in the scrap feedstock M depending upon the particular composition of the feedstock. The scrap flakes F typically have sizes in the range of 0.10 to 0.30 inches for maximum width dimension and 0.02 to 0.06 inches for thickness dimension, although the as-collected scrap material can be comminuted to other flake sizes and shapes for solid state pulverization pursuant to the invention. A conventional scrap chopping machine of the grinder type can be used in practicing the invention, although other comminuting machines also can be used to this end.

The comminuted sorted or unsorted, commingled scrap flakes F are supplied as feedstock to a twin-screw extruder 10 shown schematically in Figure 1 to effect solid state shear pulverization of the flake scrap material in accordance with the invention. Comminuted unsorted, commingled scrap material from different sources can be fed to the extruder as feedstock. Still further, comminuted unsorted, commingled scrap material from different sources can be fed to the extruder as distinct, sequential feedstocks. Alternately, comminuted flake scrap material that is sorted so as to have a single polymer composition or generic type (e.g. HDPE, PP, etc.) can be fed to the extruder as feedstock.

The extruder 10 includes a feeder 12 for receiving the polymeric flake scrap feedstock M for transport by the twin screws 14 (only one shown) through the extruder barrel zones S1-SN TOTAL where SN TOTAL corresponds to the total number of extruder zones. The first zone S1 is a material feed zone communicated to the feeder 12. Extruder barrel zones S2-SN each may be heated by external electric heater bands BH on the extruder barrel 16, depending on the composition and degree of crystallization of the scrap components being fed to the extruder. Zones S2-SN are followed by zones SN+1 to SN TOTAL that are cooled by coolant manifold bands or

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collars BC on the extruder barrel 16 (with the exception of certain barrel throttle zones not shown in Figure 1 which are air cooled)..

Alternately, the extruder barrel zones S2-SN are cooled by coolant bands similar to coolant bands BC followed by the aforementioned cooled downstream extruder zones SN+1-SN TOTAL such that only frictional heating of the scrap material occurs in the extruder. Use of the cooled extruder barrel zones S2-SN TOTAL is preferred to facilitate temperature control of the scrap material during the pulverization operation and to reduce degradation of the polymer and dye or colorant used with the polymers. Energy consumption during the pulverization operation also is reduced compared to conventional scrap batch grinding processes.

In Figure 1, the flake scrap material feedstock M is supplied by the feeder 12 to the twin-screw extruder 10 having side-by-side, intermeshing, co-rotating screws 14 (only one shown in Fig. 1) in the extrusion barrel 16, although the invention generally envisions using one or more extruder screws to achieve solid state pulverization. The screws 14 are rotated in the same direction by drive motor 18 through a gearbox (not shown) to transport the scrap material along the length of the screws 14 and subject the scrap feedstock to temperature and pressure conditions for a given scrap feed rate effective to achieve solid state shear pulverization thereof (without melting of the polymers) and in-situ compatibilization of any thermodynamically incompatible polymers present in the scrap feedstock. The solid state pulverization avoids melting of the polymeric scrap material in the feedstock but can involve softening of one or more of the polymers in the feedstock.

Uniform pulverized polymeric particulates P (e.g. powder) are discharged at the open (dieless) discharge end 16a of the extruder barrel 16. The pulverized

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particulates P exhibit differential scanning calorimetry (DSC) and electron spin resonance spectroscopy (ESR) characteristics different from those exhibited by the unpulverized flake scrap feedstock M. Moreover, increased tensile strengths and lack of delamination upon breaking of testing specimens molded from the pulverized powder are further indicative of in-situ polymer compatibilization.

10       The morphological transformation of the scrap feedstock M, whether of the sorted or unsorted, commingled type, as it is transported through the typical zones S1-SN of the extruder barrel 16 and subjected to solid state shear pulverization therein is illustrated in Figure 2. For purposes of illustration and not limitation, feedstock M is shown in Figure 2 including three (3) different flake colors by the three different flake surface shadings shown (i.e. cross-hatched, dotted, and clear flakes).

20       Referring to Figure 2, the scrap flakes F are transformed first to large fluff having a particle morphology characterized as fibrous, easily-peeled, laminated particles and substantially homogeneous light color based on a dominant color of a particular scrap component in the feedstock M. During the pulverization process, the flakes F appear to be sheared and deformed into elongated strands. These strands break to form roughly spherical (rounded) particles. The large fluff is further transformed to coarse fluff, fluff, coarse powder and then powder as illustrated in Figure 2 in  
30       dependence on the location of the scrap feedstock along the length of the extrusion screws 14. Both the fluff and powder products of solid state pulverization can find use as feedstock in melt and other polymer processing techniques to form shaped articles of manufacture.

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As is apparent, the size of the scrap particulates gets smaller as the feedstock passes along the length of the screws 14. The fragmentation of the feedstock that occurs during pulverization produces powder particles that can be smaller than any of the clearances between barrel and screws of the extruder 10. Moreover, although not apparent from Figure 2, the color of the scrap particulates typically gets slightly lighter as the feedstock is transported along the length of the screws 14 such that the pulverized powder P has a substantially homogeneous light pastel color tone based on a dominant color of a scrap component in the feedstock. For example, if the feedstock M includes white, blue and orange colored particulate flakes F in equal proportions, the color of the large fluff will be a homogeneous light pastel orange color tone. If the feedstock M includes white, red, blue and yellow colored particulate flakes F in equal proportions, the color of the large fluff will be a homogeneous light pastel rose color tone. As will be explained, the substantially homogeneous light pastel color of the fine recycled polymeric powder persists through subsequent melt processing (e.g. injection molding) to yield a product having a substantially homogeneous, single light pastel color tone without color streaking or marbling.

The pulverized particulates P (powder particles) have a rounded shape and smooth surfaces which are suggestive of a solid state shearing phenomenon. The powder particles typically have a size of less than about 500 microns. The size range of a particular recycled polymeric powder produced by the invention will depend on composition of the feedstock M, the degree of crystallization of the scrap components of the feedstock M, screw configuration, and the pulverization parameters employed, such as pulverization temperatures, pressures,

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screw rpm, and feed rates of the feedstock M through the extruder 10.

Importantly, as mentioned, any thermodynamically incompatible polymers present in the feedstock M are in-situ compatibilized during solid state shear pulverization in the extruder 10 as evidenced by production of pulverized polymeric particulates P at the discharge end of the extruder that exhibit DSC and ESR characteristics different from those of the unpulverized flake scrap feedstock M of the same composition. Moreover, the recycled particulates exhibit chemical changes (enhanced reactivity) as compared to the unpulverized flake scrap feedstock M as indicated by changes in the ESR spectra showing presence of free radicals.

In practicing the invention to achieve solid state shear pulverization of the scrap feedstock M in the manner described hereabove, the twin-screw extruder 10 can comprise for purposes of illustration and not limitation the model ZE 40A twin-screw extruder (L/D 40.5) manufactured by Hermann Berstorff Maschinenbau GmbH, PO Box 629, D-3000 Hanover 1 Germany. The ZE 40A twin-screw extruder includes twin, side-by-side intermeshing screws having a modular construction wherein each screw includes a series of standard screw elements mounted end-to-end in a selected sequence on a continuous screw shaft. The screw elements or stages include fluted transport elements, kneading or shearing elements, and spacer elements mounted in a selected sequence on the continuous shaft to accommodate variations in pulverization parameters and feedstock compositions, as necessary. In particular, the screws 14 can be modified by selection of appropriate transport elements, kneading or shearing elements, and spacer elements and their sequential arrangement and element lengths along the axis of the screw shaft.

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Moreover, the ZE 40A twin screw extruder includes an extruder barrel 16 having a modular construction wherein a series of standard barrel sections or zones are secured (e.g. bolted) together end-to-end to provide a continuous extruder barrel 16. The barrel sections are adapted to have the electrical heating bands BH or the liquid cooling manifold bands BC mounted thereon for controlling temperature of the extruded material.

10 The ZE 40A twin screw extruder is described in detail in product brochure entitled "Berstorff High Performance Twin Screw Extruders ZE \* ZE-A \* ZE/ZE-A" available from Berstorff Corporation, 8200 Arrowridge Blvd., PO Box 240357, Charlotte, North Carolina, 28224. In addition, twin screw extruders of this general type are described and shown in US Patent 4 607 797, the teachings of which are incorporated herein by reference.

20 Figure 3 illustrates the extruder barrel and screw configuration used in practicing the aforementioned embodiment of the invention wherein the scrap material is heated in the zones 2-4 by the electrical heating bands BH mounted on those barrel sections or zones. Zones 5-9 are cooled by the coolant manifold bands BC. Barrel throttle zones 12, 13 and 14 are air cooled.

30 Figure 4 illustrates the extruder barrel and screw configuration used in practicing the aforementioned embodiment of the invention wherein the scrap material is heated only by frictional engagement with the twin screws 14. In Figure 4, the barrel sections or zones 2-9 are cooled by coolant bands BC disposed thereon. Barrel throttle zones 12, 13 and 14 are air cooled.

Only one screw 14 is shown in Figures 3 and 4; however, the other screw 14 is identical and in intermeshing relation thereto as is known and shown in the aforementioned US Patent 4 607 797.

The invention is not to be construed as limited to the particular type or sequence of screw elements and



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barrel sections shown in Figures 3-4 or the particular twin-screw extruder described. Extruder barrel and extruder screw configurations used in practicing the invention can be different from those shown and may depend upon the composition and degree of crystallization of scrap components of the feedstock M as well as other factors in order to achieve solid state shear pulverization and in-situ polymer compatibilization of the feedstock M to pulverized particulates in accordance with the invention.

The scrap feedstock M is subjected to solid state shear pulverization in the extruder 10 in a once-through operation by rotation of the twin screws 14 in the same directions by motor 18 so as to transport the feedstock M along the length of the screws 14 and subject the feedstock M to temperature and pressure conditions (for a given feedstock feed rate through the extruder) effective to achieve solid state shear pulverization (without melting) of the flake-shaped scrap particulates and in-situ polymer compatibilization of thermodynamically incompatible polymers present in the feedstock M. The solid state pulverization avoids melting of the polymeric scrap material in the feedstock but can involve softening of one or more of the polymers in the feedstock.

With respect to the temperature parameter at a given feedstock feed rate, the feedstock M can be heated at extruder barrel sections or zones 2-4 depending upon the composition of the feedstock M followed by cooling in all subsequent barrel zones of the extruder to maintain proper temperature control for solid state pulverization, in-situ polymer compatibilization, if necessary, and production of desired powder size. Heating of the feedstock M in barrel zones 2-4 is achieved by energization of the electrical heater bands or collars BH mounted on the barrel zones 2-4. Cooling

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of the feedstock at barrel zones 5-9 is achieved by individual, coolant manifold bands or collars BC mounted on each barrel zone, and supplied (from a common coolant manifold conduit not shown) with a water/glycol mixture that is chilled to nominally 35°F and pumped by a conventional pump (not shown) at a selected flow rate through the manifold bands BC. Barrel throttle zones 12, 13 and 14 are air cooled by ambient air.

10 Heating of the scrap feedstock M in barrel zones 2-4 may be effected when pulverizing a feedstock M having a large percentage of amorphous or crystalline scrap polymeric materials. Pulverization occurs in cooled barrel zones where the kneading or shearing elements KE are located. Polymeric material is transported to the kneading or shearing elements by the fluted transport elements appropriately positioned along the lengths of the screws to this end. The screw elements in each zone should be selected appropriately. Spacer elements are used to provide the appropriate screw length for the  
20 extruder barrel used.

Preferably, the scrap feedstock M is subjected to only frictional heating in barrel zones 2-4 by engagement with the rotating screws 14. That is, solid state pulverization of the scrap feedstock in barrel zones 2-4 is conducted without heating of the feedstock by external heater bands BH. Instead, all barrel zones 2-9, are cooled by coolant manifold barrels or collars BC disposed on the extruder barrel. Pulverization occurs in cooled barrel zones where the kneading or shearing  
30 elements KE are located. This heatless pulverization of the scrap feedstock M is advantageous to reduce heat degradation of the polymers and dyes (or other colorants) present with the polymers. Energy consumption during the pulverization operation also is substantially reduced.

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In general, the temperature of the scrap feedstock M is maintained at a level below the melting temperature of the individual scrap components or constituents in the feedstock M at all barrel zones of the extruder 10 regardless of whether barrel zones 2-4 are heated or cooled. In this way, solid state shear pulverization can be conducted in the solid state at all stages of the extruder 10. The temperature of the feedstock M at each stage of the extruder 10 will depend on the particular feedstock composition being pulverized.

For example, scrap feedstock temperatures to achieve solid state shear pulverization will depend upon the ratio of various polymers in the feedstock M. In particular, the feedstock temperature is adjusted to accommodate a relatively high proportion of amorphous, polymeric material (e.g. PS) in the feedstock M. For high proportions (e.g. 70 weight %) of amorphous scrap materials, the temperature is lower as compared to the feedstock temperatures used to pulverize a feedstock M having a high proportion of crystalline scrap materials (e.g. PP). The feedstock temperature needed for solid state shear pulverization of the feedstock M to powder can be related to glass transition temperature and/or melting temperature for particular feedstock compositions by suitable adjustment to heat output of barrel heater bands BH, if used, and to the coolant flow rate through the extruder coolant manifold bands BC.

Illustrative extruder barrel temperatures at various stages of the extruder 10 are described in certain Examples set forth below for various feedstock compositions for purposes of illustration and not limitation.

With respect to the pulverization pressure and shear rate, the pressure and shear rate are controlled for a given screw design by the torque and rotational speed (rpm) of the motor 18 rotating the screws 14. The pressure and shear forces on the scrap feedstock M are

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effective for given feedstock temperatures and feed rates to achieve solid state shear pulverization of the flake-shaped scrap particulates in the manner described hereabove to achieve formation of the rounded powder and in-situ polymer compatibilization. The feedstock pressures and shear rates to achieve solid state shear pulverization will depend upon the ratio of various polymers in feedstock M. For high proportions (e.g. 70 weight %) of amorphous scrap materials, the energy consumption is higher as compared to the feedstock pressures used to pulverize a feedstock M having a high proportion of crystalline scrap materials. The feedstock pressures needed for solid state shear pulverization of the feedstock M to powder can be determined empirically for particular feedstock compositions by suitable adjustment to the torque and rpm of screw drive motor 16 at given feedstock temperatures.

The through-put (e.g. pounds per hour) of the scrap feedstock M through the extruder is controlled in conjunction with temperature and pressure to achieve solid state shear pulverization of the flake-shaped scrap particulates. The through-put for given feedstock temperature/pressure parameters needed for solid state shear pulverization of the feedstock M to fine powder can be determined empirically for particular feedstock compositions by suitable adjustment to the rpm of drive motor 16 for screws 14.

Illustrative through-puts of the scrap feedstock M through the extruder are described in certain Examples set forth below for various feedstock compositions and temperature/pressure parameters for purposes of illustration and not limitation.

By proper selection of the pulverization temperatures, pressures, through-puts, and screw configuration and rpm, pulverized polymeric particulates P (e.g. powder) are discharged at the open (dieless)

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discharge end 16a (i.e. open end without the usual extrusion die) of the extruder barrel 16. The particulates P typically are discharged onto a conventional endless conveyor (not shown) or other cooling/transport device, such as a spiral cooling device, capable of cooling powders and transporting powders to a storage location. During transport on the conveyor, the recycled pulverized powder can be allowed to cool to ambient temperature preferably in an atmosphere inert to the powder, such as a nitrogen blanket. Use of an inert blanket avoids oxidation of the pulverized powder.

The recycled pulverized particulates P of the invention are made without the need for a compatibilizing agent despite the presence of two or more thermodynamically incompatible polymeric scrap materials in the initial feedstock M. Moreover, the recycled pulverized particulates P of the invention can be directly processed (i.e. without pelletization) by conventional rotational molding, blow molding, extrusion (extruded PVC pipe and profiles), spray coating and other melt processing techniques requiring powder feedstock. Further, the recycled particulates P exhibit enhanced reactivity as compared to the unpulverized scrap feedstock M as measured by ESR. If produced from mixed-color scrap feedstock M, the recycled polymeric particulates, pulverized and as-melt processed, exhibit a substantially homogeneous light pastel color based on a dominant color of a scrap component in the feedstock M, although a color adjustment can be made, if desired, by suitable pigment additive to the feedstock and/or pulverized particulates.

As the Examples below will illustrate, injection molded articles of manufacture produced from the recycled, pulverized particulates P of the present invention exhibit mechanical properties generally superior to those exhibited by like-molded flake scrap

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polymer feedstock of the same composition and also a substantially homogeneous color characterized by the absence of color streaking or marbling, despite the powder having originated from mixed-color scrap feedstock. The homogeneous color of molded articles produced from the pulverized particulates is quite surprising and unexpected given the mixed-color nature of the initial feedstock M. The Examples set forth herebelow illustrate these advantageous features of the invention.

Importantly, the recycled, pulverized particulates P of the invention can be used in molding a plurality of articles that exhibit substantially the same homogeneous pastel color from one molded article to the next as illustrated in the following Examples. In contrast, a mixture of flake scrap of like composition and mixed color produce molded articles exhibiting inconsistent colors from one molded article to the next.

The recycled particulates P of the invention also can be used in conventional coating procedures wherein powdered coating feedstock is used. For example, the recycled powder can be used as feedstock in coating processes, such as thermal spray, to produce homogeneously colored, thin coatings on various substrates.

The following Examples are offered for purposes of illustrating the invention in greater detail without in any way limiting the invention. These Examples involve sorted, washed, and chopped polymeric post-consumer flake scrap material obtained from several recycling sources throughout the United States; namely, HDPE and chopped LDPE flake scrap was obtained from St. Joseph Plastics in St. Joseph, MO.; chopped PP scrap flake was obtained from SEI Recycling in Burlington, WI.; and chopped PET scrap flake was obtained from Plastic Recyclers Southeast, Inc. in Athens, AL. These scrap

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materials were obtained in 700 pound quantities. PVC scrap in flake form was obtained from Oxychem Corp. in Dallas, Texas. PS scrap in flake form was obtained from Maine Plastics in North Chicago, IL.

10 Various proportions of these scrap materials were dry blended in order to simulate typical scrap feedstock compositions that would be encountered in the recycling of plastic scrap from municipal collection centers and industrial collection centers. Binary, ternary, and quaternary blends of different polymeric scrap materials were simulated (See TABLE II below). The scrap components of the dry blends are based on weight percentages of the components involved.

20 Various mechanical property test specimens were prepared by injection molding 1) the as-received individual flake scrap materials and their blends (referred to as "Flake" in Table II below) and 2) the pulverized powder P of the invention (referred to as "Pulverized" or "Pulv" in Table II below). A 4-cavity MUD family mold was used for injection molding with a 1 oz. Battenfield injection molding machine. Specimens were of the standard ASTM "dog bone" shape, approximately 5 inches long and 0.125 inch thick. Izod bars (2.5 inches long and 0.125 inch thick) and five-inch heat distortion temperature (HDT) bars were also similarly molded.

30 The injection molded specimens were used in the indicated ASTM tests of tensile strength/elongation (D638), notched Izod impact strength (D256), heat distortion temperature measurements (D648), and Shore D hardness (D2240). Tensile strengths are reported as ultimate tensile strengths for most specimens (yield strength being reported for the LDPE and PP specimens). Tensile tests were measured with an Instron test machine (crosshead speed = 2 inches/minute, gauge length = 2.25

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inches, strain rate = 0.9 in./in.-min.). Elongations are reported at the breaking point.

#### EXAMPLE 1

The aforementioned chopped PP scrap flakes (obtained from SEI Recycling in Burlington, WI) were solid state pulverized using the aforementioned ZE 40A twin screw extruder. A sample of the scrap flakes is shown in Figure 5A prior to solid state pulverization in accordance with the invention. Although the flakes all comprised PP, they were of mixed-colors; i.e. white and taupe flakes, red flakes, yellow flakes, blue flakes and a small amount of black flakes.

The PP scrap flake feedstock was solid state pulverized pursuant to the embodiments of the invention described above wherein in one trial, there was no external heating of the scrap flakes in the extruder (i.e. heatless embodiment) and wherein the heater bands BH were energized to heat the scrap flakes in barrel zones 2-4 (i.e. externally heated embodiment).

TABLE I below sets forth the temperature, rpm (for motor 18), and scrap through-put employed in the trials to solid state pulverize the PP scrap flake feedstock. Both the extruder control set temperature and measured temperature of the scrap in the extruder are shown in TABLE I. As is apparent, the trial wherein external heating ("No heat") involved higher scrap temperatures, lower motor rpm and higher scrap through-put than when heater bands BH were used to heat the scrap flakes ("w/heat"). Pressure in the extruder was not measured in the Examples.

With respect to the configuration of the twin screws 14, the heatless trial used the screw configuration shown in Figure 4. The externally heated trial used the screw configuration shown in Figure 3.



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Figure 5A shows the flake scrap feedstock and an injection molded specimen made therefrom. Figure 5B shows the pulverized powder of the invention made with external heating and an injection molded specimen made therefrom.

Figures 5A and 5B illustrate that the pulverized powder of the invention made without external heating comprised fine powder particles having a homogeneous light mauve color as opposed to the mixed-color scrap flakes. Moreover, the injection molded pulverized powder specimen exhibited a substantially homogeneous, slightly more intense mauve color without color streaking or marbleizing. In contrast, the injection molded scrap flakes exhibited a non-uniform color with color streaking and marbleizing throughout the specimen, Figure 5A.

The pulverized powder of the invention made without external heating of the extruder barrel zones (all barrel zones cooled) exhibited a similar morphology and a uniform purple/cranberry color. The injection molded shape made from this powder exhibited a substantially homogeneous, slightly more intense cranberry color without color streaking or marbleizing.

The mechanical properties measured from the aforementioned injection molded dog bone specimens made from the PP scrap flake feedstock and PP pulverized powder are set forth in TABLE II. These mechanical properties are discussed below.

#### EXAMPLE 2

The aforementioned chopped HDPE scrap flakes (obtained from St. Joseph's Plastics St. Joseph, Mo.) were solid state pulverized using the aforementioned ZE 40A twin screw extruder. Although the flakes all comprised HDPE, they were of mixed-colors; i.e. white and taupe flakes, green flakes, blue flakes, and small amount of yellow flakes.

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The HDPE scrap flake feedstock was solid state pulverized pursuant to the embodiments of the invention described above wherein in one trial, there was no external heating of the scrap flakes in the extruder (i.e. heatless embodiment) and wherein the heater bands BH were energized to heat the scrap flakes in barrel zones 2-4 (i.e. externally heated embodiment).

10       TABLE I below sets forth the temperature, rpm (for motor 18), and through-put employed in the trials to solid state pulverize the HDPE scrap flake feedstock. The heatless trial used the screw configuration shown in Figure 4. The externally heated trial used the screw configuration shown in Figure 3.

The recycled pulverized HDPE powder made from the scrap flake feedstock without external heating exhibited a light blue-green color and powder morphology. The pulverized HDPE powder produced with external heating was similar in morphology but had a somewhat lighter green-blue color.

20       The injection molded pulverized HDPE powder specimens exhibited a substantially homogeneous, slightly more intense colors without color streaking or marbling. In contrast, the injection molded scrap HDPE flakes exhibited a uniform whitest color.

The mechanical properties measured from the aforementioned injection molded dog bone specimens made from the HDPE scrap flake feedstock and HDPE pulverized powder are set forth in TABLE II and are discussed below.

30       EXAMPLE 3

The aforementioned chopped LDPE scrap flakes (obtained from St. Joseph's Plastics St. Joseph, Mo.) were solid state pulverized using the aforementioned ZE 40A twin screw extruder. Although the flakes all comprised

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LDPE, they were of mixed-colors; i.e. white flakes, beige flakes, red flakes, and blue flakes.

The LDPE scrap flake feedstock was solid state pulverized pursuant to the embodiments of the invention described above wherein in one trial, there was no external heating of the scrap flakes in the extruder (i.e. heatless embodiment) and wherein the heater bands BH were energized to heat the scrap flakes in barrel zones 2-4 (i.e. externally heated embodiment).

10        TABLE I below sets forth the temperature, rpm (for motor 18), and through-put employed in the trials to solid state pulverize the LDPE scrap flake feedstock. The heatless trial used the screw configuration shown in Figure 4. The externally heated trial used the screw configuration shown in Figure 3.

20        The recycled pulverized LDPE powder made from the scrap flakes without external heating exhibited a uniform light beige color and powder morphology. The pulverized LDPE powder made using external extruder barrel heating was a homogeneous light orange-beige color with similar morphology.

The injection molded pulverized LDPE powder specimen exhibited a substantially homogeneous, slightly more intense beige color (heatless), or orange-beige color (with external barrel heating), without color streaking or marbleizing. The injection molded scrap LDPE flakes exhibited a uniform burnt orange color.

30        The mechanical properties measured from the aforementioned injection molded dog bone specimens made from the LDPE scrap flake feedstock and LDPE pulverized powder are set forth in TABLE II and are discussed below.

#### EXAMPLE 4

The aforementioned chopped HDPE, LDPE and PP scrap flakes were mixed to simulate a scrap feedstock compris-

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ing 60 weight % HDPE, 30 weight % LDPE and 10 weight % PP flakes. The feedstock was solid state pulverized using the aforementioned ZE 40A twin screw extruder. The HDPE/LDPE/PP scrap flake feedstock comprised mixed-colors; i.e. white flakes, red flakes, sky blue flakes, and orange flakes. Figure 6A illustrates the HDPE/LDPE/PP scrap flake feedstock.

10 The HDPE/LDPE/PP scrap flake feedstock was solid state pulverized pursuant to the embodiment of the invention described above wherein the heater bands BH were energized to heat the scrap flakes in barrel zones 2-4 (i.e. externally heated embodiment). The screw configuration shown in Figure 3 was used.

The recycled pulverized HDPE/LDPE/PP powder made from the scrap flake feedstock is shown in Figure 6B. The pulverized powder was a homogeneous light pastel rose color.

20 The injection molded pulverized HDPE/LDPE/PP powder specimen exhibited a substantially homogeneous, slightly more intense pastel rose color without color streaking or marbleizing, Figure 6B. In contrast, the injection molded scrap HDPE/LDPE/PP flake feedstock exhibited a non-uniform, streaky beige/pink color, Figure 6A.

The mechanical properties measured from the aforementioned injection molded dog bone specimens made from the HDPE/LDPE/PP scrap flake feedstock and HDPE/LDPE/PP pulverized powder are set forth in TABLE II and are discussed below.

#### EXAMPLE 5

30 The aforementioned chopped HDPE and PP scrap flakes were mixed to simulate a scrap feedstock comprising 70 weight % HDPE and 30 weight % PP flakes. The feedstock was solid state pulverized using the aforementioned ZE 40A twin screw extruder. The HDPE/PP scrap flake feedstock comprised mixed-colors; i.e. white and taupe

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flakes, green flakes, red flakes, dark blue flakes, and small amounts of yellow flakes and black flakes. Figure 7A illustrates the HDPE/PP scrap flake feedstock.

The HDPE/PP scrap flake feedstock was solid state pulverized pursuant to the embodiment of the invention described above wherein the heater bands BH were energized to heat the scrap flakes in barrel zones 2-4 (i.e. externally heated embodiment). The screw configuration shown in Figure 3 was used.

10       The recycled pulverized HDPE/PP powder made from the scrap flake feedstock is shown in Figure 7B. The pulverized powder was a homogeneous light pastel rose color.

Moreover, the injection molded pulverized HDPE/PP powder specimen exhibited a substantially homogeneous, slightly more intense pastel rose color without color streaking or marbleizing, Figure 7B. In contrast, the injection molded scrap HDPE/PP flake feedstock exhibited a non-uniform, streaky green/pink color, Figure 7A.

20       The mechanical properties measured from the aforementioned injection molded dog bone specimens made from the HDPE/PP scrap flake feedstock and HDPE/PP pulverized powder are set forth in TABLE II and are discussed below.

#### EXAMPLE 6

30       The aforementioned chopped HDPE and PP scrap flakes were mixed to simulate a scrap feedstock comprising 90 weight % HDPE and 10 weight % PP flakes. The feedstock was solid state pulverized using the aforementioned ZE 40A twin screw extruder. The HDPE/PP scrap flake feedstock comprised mixed-colors; i.e. white and taupe flakes, green flakes, red flakes, dark blue flakes, and small amounts of yellow flakes. The quantity of red flakes present was less than that in scrap flake

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feedstock of Example 4. Figure 8A illustrates the HDPE/PP scrap flake feedstock.

The HDPE/PP scrap flake feedstock was solid state pulverized pursuant to the embodiments of the invention described above wherein the heater bands BH were energized to heat the scrap flakes in barrel zones 2-4 (i.e. externally heated embodiment). The screw configuration shown in Figure 3 was used.

10 The recycled pulverized HDPE/PP powder made from the scrap flake feedstock is shown in Figure 8B. The pulverized powder was a homogeneous light pastel green color.

Moreover, the injection molded pulverized HDPE/PP powder shape exhibited a substantially homogeneous, slightly more intense pastel green color without color streaking or marbleizing, Figure 8A. In contrast, the injection molded scrap HDPE/PP flakes exhibited a non-uniform, streaky green/pink color, Figure 8A.

20 The mechanical properties measured from the aforementioned injection molded dog bone specimens made from the HDPE/PP scrap flake feedstock and HDPE/PP pulverized powder are set forth in TABLE II and are discussed below.

#### EXAMPLE 7

30 The aforementioned chopped HDPE and LDPE scrap flakes were mixed to simulate a scrap feedstock comprising 40 weight % HDPE and 60 weight % LDPE flakes. The feedstock was solid state pulverized using the aforementioned ZE 40A twin screw extruder. The HDPE/PP scrap flake feedstock comprised mixed-colors; i.e. white flakes, green flakes, blue flakes, red flakes, and small amounts of yellow flakes.

The HDPE/LDPE scrap flake feedstock was solid state pulverized pursuant to the embodiments of the invention

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described above wherein all barrel zones were cooled by collars BC.

TABLE I below sets forth the temperature, rpm (for motor 18), and through-put employed in the trials to solid state pulverize the feedstock. The screw configuration shown in Figure 4 was used.

The recycled pulverized HDPE/LDPE powder made from the scrap flake feedstock was a homogeneous light pastel peach color.

10        Moreover, the injection molded pulverized HDPE/LDPE powder shape exhibited a substantially homogeneous, slightly more intense light pastel peach color without color streaking or marbleizing. In contrast, the injection molded scrap HDPE/LDPE flakes exhibited a non-uniform, streaky beige/brown color.

20        The mechanical properties measured from the aforementioned injection molded dog bone specimens made from the HDPE/LDPE scrap flake feedstock and HDPE/LDPE pulverized powder are set forth in TABLE II and are discussed below.

TABLE I

Plastic	S = Set, A = Actual														Barrel zone temperature, °C										Motor RPM	Color	
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	Powder	Injection molded samples											
PP	A	-	19	87	54	31	52	38	24	18		52	43	31	150	light mauve	more intense (deeper) mauve										
No heat	S	-	0	0	0	0	0	0	0	0		0	0	0													
PP	A	19	224	220	221	152	92	85	54	33		179	99	60	40	cran-berry	more intense (deeper) cranberry										
W/heat	S	0	220	220	220	0	0	0	0	0		0	0	0													
HDPE	A	-	14	71	32	19	32	30	18	15		0	0	0	80	light green/blue	more intense (deeper) greenblue										
No heat	S	-	0	0	0	0	0	0	0	0		0	0	0													
HDPE	A	18	120	120	122	117	88	74	47	29		119	91	51	40	blue/green	more intense (deeper) bluegreen										
W/heat	S	0	125	125	125	0	0	0	0	0		0	0	0													
LDPE	A	-	26	67	47	27	43	29	21	17		40	36	28	80	light beige	more intense (deeper) beige										
No heat	S	-	0	0	0	0	0	0	0	0		0	0	0													
LDPE	A	19	159	158	178	139	80	60	44	27		167	97	55	40	orange/beige	more intense (deeper) orange/beige										
W/heat	S	0	175	175	175	0	0	0	0	0		0	0	0													
HDPE/LDPE A 40/60	A	19	175	179	159	52	30	21	19	17		107	27	31	20	light peach	light peach										
W/heat	S	0	175	175	160	0	0	0	0	0		0	0	0													
HDPE/LDPE A 40/60	A	-	75	21	26	20	21	21	14	15		22	22	25	113	light peach	light peach										
No heat	S	-	0	0	0	0	0	0	0	0		0	0	0													

PP = PolypropyleneHDPE = High Density PolyethyleneLDPE = Low Density Polyethylene

PP = Polypropylene      HDPE = High Density Polyethylene      LDPE = Low Density Polyethylene



## OTHER EXAMPLES

The aforementioned chopped scrap flakes alone or mixed together were used to simulate other scrap feedstocks listed in TABLE II below. Each simulated scrap flake feedstock was solid state pulverized using the aforementioned ZE 40A twin screw extruder. The scrap flake feedstocks of all Examples comprised mixed-colors in myriad combinations of color.

10 The scrap flake feedstocks were solid state pulverized pursuant to the embodiments of the invention described above wherein in one trial, there was no external heating of the scrap flakes in the extruder (i.e. heatless embodiment) and wherein the heater bands BH were energized to heat the scrap flakes in barrel zones 2-4 (i.e. externally heated embodiment). The heatless trials are designated "No heat pulv" in TABLE II and used the screw configuration shown in Figure 4. The externally heated trials are designated by the absence of "No heat pulv" and used the screw  
20 configuration shown in Figure 3. In Table II, Examples 1-7 are identified by the example number in parenthesis in the left-hand column.

The temperature, rpm (for motor 18), and through-put employed in the trials were selected empirically to achieve solid state pulverization. The temperature, motor rpm and through-put used were generally in the same ranges as those described above in Examples 1-3 and 7 for the heatless and externally heated trials.

30 In Other Examples, molded specimens prepared from the pulverized powder of the invention exhibited a surprisingly and extremely uniform light pastel color based on a dominant color scrap component in the feedstock. The achievement of the uniform, light pastel color was surprising and unexpected, given the mixed-color nature of most of the initial scrap flake feedstocks used.

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In contrast, the color of molded specimens prepared from the as-received flake feedstocks exhibited a streaking or marbleizing effect with the presence of light and dark color swirling patterns readily apparent.

Specimens molded from pulverized feedstocks including both PET and PVC were the only exceptions to achievement of a light pastel molded color. The molded specimens containing both PET and PVC exhibited a homogenous color that was darker than the pastel color tone observed with the other specimens containing either PET or PVC.

All of the specimens molded from the pulverized powder of the invention exhibited a very smooth, homogenous colored surface. In contrast, the specimens molded from the as-received flake feedstocks exhibited a non-uniform, streaky surface.

It is noteworthy that even though some of the scrap feedstock set forth in Examples 7-25 comprised a plurality of polyolefins (e.g. HDPE, LDPE, PP) that are mutually thermodynamically incompatible, these feedstocks were nevertheless successfully injection molded.

TABLE II summarizes the measured mechanical properties and thermal properties specimens of Examples 1-7 and the aforementioned Other Examples molded from as-received flake feedstocks (Flake) and pulverized powder P of the invention (Pulverized or Pulv).

TABLE II

Materials	Tensile Properties			Notched Izod Impact	HDT	Shore D
	Yield PSI	Ultimate PSI	% Elong	Ft-Lb/In	Deg C 66 PSI	
PP Flake (1)	4840		330	0.7	102	72
PP Pulverized (1)	4730		100	0.8	93	72
PP--No heat pulv (1)	5060		300	0.6	97	74
HDPE Flake (2)		3240	12	0.5	60	63
HDPE Pulverized (2)		2870	7.5	0.5	60	63
HDPE--No heat pulv (2)		3400	14	0.5	57	57
LDPE Flake (3)	1980		33	0.8	49	56
LDPE Pulverized (3)	2060		60	0.7	50	57
LDPE--No heat pulv (3)	2060		32	0.7	49	57
HDPE/LDPE/PP 60/30/10 Flake (4)		2680	9	0.4	58	62
HDPE/LDPE/PP 60/30/10 Pulv (4)		2970	9	0.4	58	64
HDPE/PP 70/30 Flake (5)		3740	11	0.4	68	67
HDPE/PP 70/30 Pulv (5)		3850	12	0.4	68	67
HDPE/PP 90/10 Flake (6)		3530	13	0.4	67	66
HDPE/PP 90/10 Pulv (6)		3480	12	0.4	63	65
HDPE/LDPE 40/60 Pulv (7)		2550	15	0.6	45	59
HDPE/PET 50/50 Flake		3710	6	0.7	70	68
HDPE/PET 50/50 Pulv		3600	4	0.5	70	69
HDPE/PET 60/40 Flake		2870	4	0.4	72	68
HDPE/PET 60/40 Pulv		2790	3	0.2	70	68
HDPE/PET 40/60 Flake		4680	7	1.1	69	71